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DATE: Saturday, April 10, 2004

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	L2	5503790.pn.	2
	L3	4092458.pn.	2
	L4	EP698462	0
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	L6	EP000698462	0
	L7	EP0698462	0
	L8	EPO698462	0
	L9	5503790.PN.	2
	L10	L9 and (grind\$4 or ground or divid\$4 or comminut\$4 or break\$4)	1
	L11	L10 and (Wash\$4 or rins\$4 or soak\$4 or alkalin\$4)	-1
	L12	L11 and (dewater\$4 or dr\$4)	1
	L13	L12 and (melt\$4 or heat\$4)	1
	L14	L13 and extru\$6	1
	L15	4092458.pn.	2

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DATE: Saturday, April 10, 2004

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	L2	5503790.pn.		2
	L3	4092458.pn.		2
	L4	EP698462		0
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	L6	EP000698462		0
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DATE: Saturday, April 10, 2004

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	L11	L10 and (Wash\$4 or rins\$4 or soak\$4 or alkalin\$4)	1
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	L14	L13 and extru\$6	1
	L15	4092458.pn.	2
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	L22	L21 and (dewater\$4 or dr\$4)	1
	L23	L22 and (melt\$4 or heat\$4)	1
	L24	L23 and extru\$6	1
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	L27	L26 and (wash\$4 or soak\$4 or rins\$4 or alkalin\$4)	. 0
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	L30	L29 and (wash\$4 or rins\$4 or soak\$4 or alkalin\$4)	1
	L31	L30 and (dewater\$4 or dri\$4)	1

Search 1	History [Transcript	Page 2 of 2
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	L37	L36 and (dewater\$4 or dr\$4)	1
	L38	L37 and (melt\$4 or heat\$4)	1
	L39	L38 and Extru\$6	1

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	L2	5503790.pn.	2
	L3	4092458.pn.	2
	L4	EP698462	0
	L5	EP0698462	0
	L6	EP000698462	0
	L7	EP0698462	0
	L8	EPO698462	0
	L9	5503790.PN.	2
	L10	L9 and (grind\$4 or ground or divid\$4 or comminut\$4 or break\$4)	1
	L11	L10 and (Wash\$4 or rins\$4 or soak\$4 or alkalin\$4)	1
	L12	L11 and (dewater\$4 or dr\$4)	1
	L13	L12 and (melt\$4 or heat\$4)	1
	L14	L13 and extru\$6	1
	L15	4092458.pn.	2
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	L19	5876644.pn.	2
	L20	L19 and (grind\$4 or ground\$4 or divid\$4 or comminut\$4 or break\$4)	2
	L21	L20 and (wash\$4 or soak\$4 or rins\$4 or alkalin\$4)	1
	L22	L21 and (dewater\$4 or dr\$4)	1
	L23	L22 and (melt\$4 or heat\$4)	1
	L24	L23 and extru\$6	1
	L25	3876485.pn.	3
	L26	L25 and (grind\$4 or ground or divid\$4 or break\$4 or comminut\$4)	1
	L27	L26 and (wash\$4 or soak\$4 or rins\$4 or alkalin\$4)	0
	L28	5225130.pn.	2
	L29	L28 and (grind\$4 or ground or divid\$4 or break\$4 or comminut\$4)	1
	L30	L29 and (wash\$4 or rins\$4 or soak\$4 or alkalin\$4)	1
	L31	L30 and (dewater\$4 or dri\$4)	1

Search 1	History T	Transcript	Page 2 of 2
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	L33	L32 and extru\$6	1
	L34	6583217.pn.	2
	L35	L34 and (grind\$4 or ground or break\$4 divid\$4 or comminut\$4)	1
	L36	L35 and (wash\$4 or rins\$4 or soak\$4 or alkalin\$4)	1
	L37	L36 and (dewater\$4 or dr\$4)	1
	L38	L37 and (melt\$4 or heat\$4)	1
	L39	L38 and Extru\$6	1
	L40	5876644.pn. and vacuum	1
	L41	5225130.pn. and vacuum	1
	L42	5503790.pn. and vacuum	2
	L43	5503790.pn. and melting with extruder	2
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	L47	5876644.pn. and vacuum	1
	L48	L47 and sort\$4	1

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L24: Entry 1 of 1 File: USPT Mar 2, 1999

DOCUMENT-IDENTIFIER: US 5876644 A

TITLE: Food quality polyester recycling

Abstract Text (1):

A method of recycling post-consumer polyester to obtain recycled polyester of sufficiently high purity to meet food packaging requirements is disclosed. The method includes cleaning comminuted pieces of post-consumer polyester to remove surface contaminants; melting the surface-cleaned post-consumer polyester pieces; extruding the post-consumer melt; blending the melt of post-consumer polyester with a melt of virgin polyester prepolymer; solidifying and pelletizing the blended melt while the virgin polyester prepolymer remains as prepolymer; and polymerizing the solid blended pellets in the solid state.

Brief Summary Text (4):

The widespread use of polymer-based products ("plastics") in consumer goods has lead to a corresponding interest in suitable methods for reclaiming or recycling as much of these products as is practical. The interest in such recycling is <u>driven</u> by several considerations. These include: environmental factors, principally occupation of landfills and other disposal issues; preservation of limited resources, because polymers are a product in the chain of petrochemicals that begins with crude oil; and economic interests, because the cost of post-consumer plastic is less related to the cost of raw materials, and instead is more dependent upon the cost of physically collecting and transporting post-consumer plastic objects to an appropriate facility for recycling. Additionally, public policy is expected to continue to emphasize environmental issues, and thus legal requirements for recycling are already in force in some jurisdictions, and are expected in others in the future.

Brief Summary Text (7):

In this regard, the term "recycling" often tends to be used relatively broadly, but actually covers a number of different ways to reuse any given material. Thus, in one sense, if a polyester bottle is used to contain a non-food product liquid rather than being directly disposed of, it has been "recycled", but the term "recycled" is rarely applied to such follow-up use of an empty container. Instead, recycling is typically identified as falling into one of three general categories, which are also recognized by the U.S. Environmental Protection Agency (EPA) and the U.S. Food and <u>Drug</u> Administration (FDA). The first is primary recycling; the use of pre-consumer industrial scrap and salvage. The second is secondary recycling, which refers to the physical reprocessing (e.g., <u>grinding</u> and re-extruding) of the polymer. The third is tertiary recycling in which a polymer is reprocessed to isolate chemical components and reuse them in manufacturing a new product.

Brief Summary Text (9):

With respect to polyester, tertiary recycling generally falls into one of several chemical processes that reduce polyester (typically polyethylene terephthalate) back into its component monomers (terephthalic acid and ethylene glycol) in some sort of depolymerizing step. Typical depolymerizing steps include methanolysis as for example practiced by Eastman Chemical Company, and Hoechst. Shell practices

another process referred to as "glycolysis," and which mixes post-consumer polyester with glycol (and potentially other compounds) to break it back down into its component precursors.

Brief Summary Text (17):

The invention meets this object with a method of recycling post-consumer polyester to obtain recycled polyester of sufficiently high purity to meet food packaging requirements which comprises cleaning comminuted pieces of post-consumer polyester to remove surface contaminants therefrom; melting the surface-cleaned post-consumer polyester pieces; extruding the post-consumer melt; blending the melt of post-consumer polyester with a melt of virgin polyester prepolymer; solidifying and pelletizing the blended melt while the virgin polyester prepolymer remains as a prepolymer; and polymerizing the solid blended pellets in the solid state.

Detailed Description Text (3):

FIG. 1 illustrates a first embodiment of the present invention. Post-consumer polyester bottles broadly designated at 10 are typically transported from consumer recycling collection sites to a comminuting facility illustrated schematically by the circles 11 that comminutes the bottles into pieces, typically flakes 12, of post-consumer polyester. At this point, because the bottles 10 have been disposed of either in the midst of other consumer trash or under other similar circumstances, they typically contain varying amounts of surface contaminants—i.e. dirt—which are removed in a washing step 13. Any appropriate combination of solvents, surfactants, water, agitation and other ingredients and techniques can be used to wash the flakes, and in a preferred embodiment the flakes are washed in a combination of basic solution (e.g. sodium hydroxide, NaOH) and detergent at a temperature of between about 20.degree. and 75.degree. C.

Detailed Description Text (4):

FIG. 1 also illustrates one of the advantages of the invention, in that the <u>washed</u> flakes can be transported as flakes to a different location for further processing. Accordingly, the <u>comminuting and washing</u> portions of the method can be carried out at separate locations as may be desired or convenient. The invention can, of course, be carried out entirely in a single location as well.

Detailed Description Text (5):

In a next step, the flakes are <u>dried in a drying</u> facility 14 at a temperature that is sufficient to remove volatile organic compounds and water without otherwise substantially changing the chemical characteristics of the <u>comminuted</u> pieces of polyester. Because <u>melting</u> points for polyester are typically about 250.degree. C., the <u>drying</u> steps should be carried out at a somewhat lower temperature, e.g. 130.degree.-200.degree. C. to accomplish the <u>drying</u> while avoiding any chemical changes. Somewhat higher temperatures can be used, however, in an inert atmosphere, such as nitrogen (N.sub.2).

Detailed Description Text (6):

The surface cleaned post-consumer polyester pieces 12 are then melted in an appropriate heating device 15 and then extruded in the melt phase using the extruder 16. If desired, the step of extruding the melt can further comprise applying a vacuum to the melt to remove additional amounts of volatile organic materials and water that remain after the drying step. The melt also can be filtered, for example, through a screen filter 17 either before extrusion or afterwards. Extrusion is preferably carried out in a two-stage single screw extruder that provides a simple and efficient extrusion step. Such extruders are well known to those of ordinary skill in the polymer and polyester arts, and will not be otherwise discussed in detail herein. Cascade extrusion can also be used, in which the first portion of the extruder melts the polyester, which then drops to a volatizing column where it is subjected to a gas sweep to further purify it, after which the swept and melted polyester is extruded. Additionally, extruders with designs other than two-stage single screw can be incorporated as may be desirable

or necessary.

Detailed Description Text (7):

In this regard, the extrusion step slightly reduces the molecular weight of the post-consumer polyester, i.e. from an intrinsic viscosity (IV) of between about 0.7 and 0.8 to an IV of between about 0.6 and 0.7. Preferably, the IV of the post-consumer melt will be within about 0.1 of the IV of the virgin polyester prepolymer with which it is blended in the next step. Although various specific techniques are used for measuring IV, it will be understood that the invention is not limited to any one particular technique, provided that the measurements are made in consistent fashion with one another.

Detailed Description Text (8):

In the next step of the invention, the <u>melt</u> of post-consumer polyester is blended with a <u>melt</u> of virgin polyester precursor resin 20, which is a relatively low IV (0.5-0.7 with 0.58-0.64 preferred) polyester typically used for later solid state polymerization. The blending step typically uses static mixers, and potentially includes filters, both of which are conventional in this art and will not otherwise be discussed in detail herein. Similarly appropriate formulations and techniques for producing virgin polyester are widely known in their art, and will not otherwise be discussed in detail herein. The basic formulation is of course, the condensation reaction between terephthalic acid and ethylene glycol followed by polymerizations to a desired molecular weight.

Detailed Description Text (9):

The blended <u>melt</u> is then solidified and pelletized to produce a plurality of blended pellets 22 in which each pellet contains the blend of the virgin polyester and the post-consumer polyester. In preferred embodiments, a greater amount of virgin polymer is blended with a lesser amount of recycled polymer, with proportions of about 75% virgin polymer and 25% post-consumer polymer being particularly favored.

Detailed Description Text (11):

FIG. 2 shows an alternative embodiment of the present invention. Where the steps and apparatus illustrated in FIG. 2 are otherwise the same as those of FIG. 1, the same reference numerals will be used with respect to FIG. 2 as were used with FIG. 1. FIG. 2 again illustrates that the bottles 10 can be collected and transported to the comminuting facility 11 where they are typically formed into flakes and washed. The flakes are again dried, melted, filtered, and extruded into pellets 25. The pellets 25 in FIG. 2 differ, however, from those in FIG. 1 in that the pellets 25 are formed entirely of post-consumer polyester while the pellets 22 in FIG. 1 each are a blend of virgin polyester and post-consumer polyester.

Detailed Description Text (12):

FIG. 2 illustrates that the steps of collection, comminuting, washing, drying, melting, extruding, and pelletizing are all carried out at an initial facility, following which the post-consumer pellets can be shipped to a second facility. In the embodiment of FIG. 2, the post-consumer pellets 25 are blended with pellets 26 that are formed entirely of virgin polyester. This produces a blend of pellets in which each individual pellet is either entirely post-consumer polyester or entirely virgin polyester. This blend of pellets is then subjected to solid state polymerization in the manner just described to produce fully polymerized polyester pellets that also meet FDA food contact requirements.

Detailed Description Text (15):

For example, Table 1 demonstrates the decrease in toluene levels from the original post-consumer polyester through solid state polymerization. As demonstrated in Table 1, if the flaked material has 1000 parts per million (ppm) by weight of toluene prior to washing, the washing step removes almost none of it. The drying step, however, reduces the toluene levels of a sample within an initial toluene

level of 1840 ppm to 1.6 ppm, a reduction ratio of over 1000. Following the extrusion step, the solid state polymerization (for a different sample) reduces the toluene level from 5.85 ppm following extrusion to 0.215 ppm following solid state polymerization, a reduction ratio of 27, resulting in a final toluene level of 0.032 ppm, or 32 parts per billion (ppb).

Detailed Description Text (16):

Table 2 demonstrates that phenyldecane can be greatly reduced by the simple steps of <u>washing and drying</u>, and Table 3 shows similar results for <u>washing and drying</u> calcium monomethyl arsenate.

Detailed Description Text (19):

In another embodiment, the invention comprises the method of recycling post-consumer polyester by reducing the intrinsic viscosity of surface-cleaned, comminuted, post-consumer polyester by between about 0.02 and 0.3 IV units by adding a component from the group consisting of water, dihydric alcohols, and polyhydric alcohols; melting the surface-cleaned post-consumer polyester pieces; extruding the post-consumer melt; blending the melt of post-consumer polyester with a melt of virgin polyester precursor; solidifying and pelletizing the blended melt; and polymerizing the solid blended pellets in the solid state.

Detailed Description Text (20):

The component can be added to the <u>melt</u> of post-consumer polyester or can be added to the surface-cleaned <u>comminuted</u> pieces of post-consumer polyester prior to the step of <u>melting</u> the post-consumer polyester. Indeed, under proper ambient conditions, the step of adding the component can comprise adding water by storing the surface-cleaned <u>comminuted</u> post-consumer polyester under ambient humidity conditions for a time sufficient for the IV to reduce by between about 0.02 and 0.03 IV units. In another embodiment, the step of adding a component comprises proactively adding ethylene glycol, another appropriate dihydric alcohol, or a polyhydric alcohol. Those polyhydric alcohols appropriate for such use will be consistent with the polymers described in title 21 of the Code of Federal Regulations, particularly Subchapter B, "Food for Human Consumption;" and Part 177 of that subchapter, "Indirect Food Additives, Polymers; Substances for Use as Basic Components of Single and Repeated Use Food Contact Surfaces." With respect to polyester, the appropriate substances are set forth at Section 177.1630 "Polyethylene Phthalate Polymers."

Detailed Description Text (21):

In yet another embodiment, the invention can comprise reducing the intrinsic viscosity of surface-cleaned comminuted post-consumer polyester by between 0.02 and 0.3 IV units by adding a component from the group consisting of water, dihydric alcohols, and polyhydric alcohols; melt extruding the surface-cleaned post-consumer polyester pieces; and then solidifying, pelletizing, and polymerizing the post-consumer polyester in the solid state. In both of these latter embodiments, the reduction in the IV helps match the IV to that of the polyester precursor with which the post-consumer polyester can be blended to improve product uniformity, and (blended or not) provides for additional residence in the solid state polymerization step which, as noted above, also serves to further purge the post-consumer polyester of any remaining organic materials.

Detailed Description Text (22):

As in the earlier described embodiments, the process of these latter embodiments can also include the <u>comminuting</u> steps, filtering of the post-consumer <u>melt</u> prior to its <u>extrusion</u>, <u>washing the comminuted</u> pieces to remove contaminants, <u>drying the washed comminuted</u> pieces, and applying a vacuum to the <u>melt being extruded</u> to help remove volatile organic materials and water.

Detailed Description Paragraph Table (1):					
TABLE 1	Initial	Final	1000	mag	Process

Sample No. Level Level Ratio Basis.sup.1 Toluene Study Incoming flake 1000 Wash 1000 Flake dryer TCM-4-78-5/6 1840 1.6 1150
1 Flake extrusion 1 1 Solid State TCM-4-99-1 5.85 (N.D.).sup.2 27 0.032
Polymerization (0.1 IV increase) Phenyldecane Wash 005-2A1 59 7 8.4 7 Flake Dryer
NEC 10934 807 1.23 656 0.011sup.1 i.e., reduction values calculated from actual amounts, but expressed proportionally as if
starting from 1000 ppm in accordance with FDA assumptionssup.2i.e., below the
0.215 detection limit
Detailed Description Paragraph Table (2):
TABLE 2 Calcium Monomethyl Arsenate Study Initial Final 1000 ppm Process Sample No. Wash 005-2A1 17.2 3
Level Level Ratio Basis Wash 005-2A1 17.2 3 5.9 3 Measure As Measure As level in level
5.9 3 Measure As Measure As level in level
in solvent (ppb) with 10 Migration Studies sheet (PPM) ppb limit of detection
8% ETOH.sup.3 -120 F TCM-4-39B 5.89 N.D. for
720 hours 50% ETOH- TCM-4-39B 5.89 N.D. 120 F for 720 hours Hot Fill 8% ETOH-120 F
TCM-4-39B 5.89 N.D. for 720 hours 50% ETOH- TCM-4-39B 5.89 N.D. 120 F for 720 hours
sup.3Ethanol, CH.sub.3 CH.sub.2 OH
Detailed Description Paragraph Table (3):
TABLE 3 Benzophenone Study
TABLE 3 Benzophenone Study 1000 Initial Final ppm Process Sample No.
Level Level Ratio Basis Wash 005-2A1 320 130
2.5 130 Flake drying NEC 10934 655 37.8 17 7.5 Solid State 101 4.79 21 0.4
Polymerization (0.1 IV) Meas- ure Measure BZ BZ level level in in sheet solvent Migration Studies (PPM) (ppb) Ratio ppb
BZ level level in in sheet solvent Migration Studies (PPM) (ppb) Ratio ppb
8% ETOH-120 F TCM-4-39B 390 21.8 17890 0.02
for 720 hours 50% ETOH- TCM-4-39B 390 1120 348 1.02 120 F for 720 hours Hot Fill 8%
ETOH-120 F TCM-4-39B 390 58 6724 0.05 for 720 hours 50% ETOH- TCM-4-39B 390 1520
257 1.39 120 F for 720 hours
Detailed Description Paragraph Table (4):
TABLE 4 Chloroform Study 1000 Initial Final
ppm Process Sample No. Level Level Ratio Basis
Incoming bottle 1000 Wash 1000 Flake dryer
TCM-4-90-2 252 2.79 90 11 Flake <u>extrusion</u> SN-1- 227 67 3 3 34D2/SM-1-34 Solid State
TCM-4-99-2 56.8 0.215 264 0.012 Polymerization /SSP (N.D.) (0.1 IV)
CLAIMS:
1. A method of recycling post-consumer polyester to obtain recycled polyester with
a residual concentration of possible food contaminants of no more than about 215
parts per billion suitable for food contact requirements, the method comprising:
cleaning comminuted pieces of post-consumer polyester to remove surface
contaminants therefrom thereby producing surface-cleaned post-consumer polyester
pieces;
melting the surface-cleaned post-consumer polyester pieces to produce a post-
consumer polyester melt;
extruding the post-consumer polyester melt to reduce the intrinsic viscosity of the
post-consumer polyester melt and remove additional contaminants;
blending the $\underline{\text{melt}}$ of post-consumer polyester with a $\underline{\text{melt}}$ of virgin polyester
prepolymer that has an intrinsic viscosity similar to the intrinsic viscosity of
the post-consumer polyester <u>melt</u> to produce a blended <u>melt</u> , and blending the <u>melts</u>
in a proportion such that one of the <u>melts</u> makes up at least about 25 percent of

the blended melt by weight;

solidifying and pelletizing the blended $\underline{\text{melt}}$ while the virgin polyester prepolymer remains as prepolymer to produce solid blended pellets; and

polymerizing the solid blended pellets in the solid state thereby forming the recycled polyester.

- 2. A recycling method according to claim 1 and further comprising a step of <u>comminuting</u> post-consumer polyester objects into small pieces of post-consumer polyester prior to cleaning the <u>comminuted</u> pieces.
- 3. A recycling method according to claim 1 and further comprising a step of filtering the post-consumer polyester $\underline{\text{melt}}$ prior to $\underline{\text{extruding}}$ the post-consumer polyester melt.
- 4. A recycling method according to claim 1 wherein cleaning the <u>comminuted</u> pieces comprises <u>washing the comminuted</u> pieces to remove surface contaminants.
- 5. A recycling method according to claim 4 wherein cleaning the <u>comminuted</u> pieces comprises <u>drying washed comminuted</u> pieces at a temperature sufficient to remove volatile organic compounds and water without otherwise changing the chemical characteristics of the comminuted pieces.
- 6. A recycling method according to claim 1 wherein $\underbrace{\text{extruding}}_{\text{extruding}}$ the post-consumer polyester $\underline{\text{melt}}$ further comprises applying a vacuum to the post-consumer polyester $\underline{\text{melt being extruded}}$ to remove volatile organic materials and water.
- 7. A recycling method according to claim 1 wherein <u>extruding</u> the post-consumer polyester <u>melt comprises extruding</u> the post-consumer polyester <u>melt</u> through a two-stage single screw extruder.
- 9. A recycling method according to claim 1 further comprising formation of the virgin polyester prepolymer from the group consisting of dicarboxylic acids, dicarboxylic esters, glycols and multifunctional alcohols prior to blending the post-consumer polyester melt with the virgin polyester prepolymer melt.
- 10. A recycling method according to claim 1 further comprising formation of the virgin polyester prepolymer from terephthalic acid and ethylene glycol prior to blending the post-consumer polyester melt with the virgin polyester prepolymer melt.
- 11. A recycling method according to claim 1 wherein blending the $\underline{\text{melts}}$ comprises blending a larger proportion of the virgin polyester prepolymer $\underline{\text{melt}}$ with a smaller proportion of the post-consumer polyester $\underline{\text{melt}}$.
- 12. A recycling method according to claim 11 comprising blending about 75 percent by weight of the virgin polyester prepolymer $\underline{\text{melt}}$ with about 25 percent by weight of the post-consumer polyester $\underline{\text{melt}}$.
- 13. A recycling method according to claim 1 wherein blending the $\underline{\text{melts}}$ comprises blending a post-consumer polyester $\underline{\text{melt}}$ with the virgin polyester prepolymer $\underline{\text{melt}}$ in which the intrinsic viscosity of the virgin polyester prepolymer $\underline{\text{melt}}$ is lower than the intrinsic viscosity of the post-consumer polyester $\underline{\text{melt}}$.
- 14. A recycling method according to claim 13 wherein the intrinsic viscosity of the virgin <u>melt</u> and the intrinsic viscosity of the post-consumer <u>melt</u> differs by about 0.1 or less.
- 15. A method of recycling polyester to obtain a recycled polyester with a residual

concentration of possible food contaminants of no more than about 215 parts per billion suitable for food contact requirements, the method comprising:

extruding a melt
viscosity of the post-consumer polyester and remove additional contaminants;

solidifying and pelletizing the post-consumer $\underline{\text{melt}}$ to produce post-consumer polyester pellets;

blending the post-consumer polyester pellets with virgin polyester prepolymer pellets that have an intrinsic viscosity similar to the intrinsic viscosity of the post-consumer pellets to produce a blend of pellets, and blending the pellets in a proportion such that other of the post-consumer or virgin pellets makes up at least about 25 percent of the blend of pellets by weight; and

polymerizing the blend of pellets in the solid state thereby forming the recycled polyester.

16. A method according to claim 15 and further comprising the steps of:

comminuting post-consumer polyester objects into small pieces, thereby producing comminuted polyester pieces; and

cleaning the <u>comminuted</u> polyester pieces to remove surface contaminants therefrom, thereby producing cleaned post-consumer polyester pieces;

both prior to extruding the cleaned post-consumer polyester pieces.

- 17. A recycling method according to claim 16 wherein cleaning the <u>comminuted</u> polyester pieces comprises <u>washing the comminuted</u> polyester pieces to remove surface contaminants.
- 18. A recycling method according to claim 16 and further comprising a step of filtering the post-consumer melt prior to extruding the post-consumer melt.
- 19. A recycling method according to claim 15 wherein <u>extruding</u> the cleaned post-consumer polyester further comprises applying a vacuum to the cleaned post-consumer polyester being <u>extruded</u> to remove volatile organic materials and water.
- 21. A recycling method according to claim 15 further comprising formation of the virgin polyester prepolymer recycled from the group consisting of dicarboxylic acids, dicarboxylic esters, glycols and multifunctional alcohols prior to blending the post-consumer polyester melt with the virgin polyester prepolymer melt.
- 22. A recycling method according to claim 15 further comprising formation of the virgin polyester prepolymer from terephthalic acid and ethylene glycol prior to blending the post-consumer polyester melt with the virgin polyester prepolymer melt.

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L33: Entry 1 of 1

File: USPT

Jul 6, 1993

DOCUMENT-IDENTIFIER: US 5225130 A

TITLE: Process for reclaiming thermally strained polyester scrap material

Abstract Text (1):

Reclaiming of polyester waste material for re-use in the production of high-quality PET plastic material comprises shredding, cleaning, sorting and postcondensing said waste materials. The shredded scrap material comprises polyester waste materials of various molecular weights and degrees of purifying. The shredded material is molten and filtered The filtered melt is either post-condensed in the molten state and then granulated or is first granulated and then subjected to a solid phase post-condensation. The granules obtained in each case are preferably fed into an extruder and extruded together with virgin raw material so as to produce a useful film or article.

Brief Summary Text (4):

German Offenlegungsschrift No. 34 33 791 discloses a process for producing plastic granules which are suitable for thermoplastic processing in an extruder, from scrap material obtained in the course of the extrusion of thermoplastic materials. The process comprises first cutting and optionally drying the material, then densifying the cut material, and finally comminuting the material into pellets which can be fed to the extruder. This known process is directed to the reclaiming of scrap material of a specific polymer, i.e., one whose mechanical, optical, and chemical properties are unchanged compared to the initial processing.

Brief Summary Text (5):

A process for the recovery of densified material from plastic films or plastic film scrap material is known from German Auslegeschrift No. 14 54 875. In this process, the films are first comminuted, then densified in an agglomeration apparatus, and transported by means of a blower equipped with a conveying line and an air separator, whereupon the densified material is comminuted in a cutting mill, to give pellets having approximately the size of the granules of the polymer raw material.

Brief Summary Text (6):

U.S. Pat. No. 3,344,091 is directed to a process for converting scrap polyester, wherein the scrap is comminuted to a certain particle size, mixed with an aromatic dicarboxylic acid ester and glycol, and heated to a temperature 25.degree. to 50.degree. C. above the boiling point of the glycol. After dissolution of the polyester and removal of the alcohol, a prepolycondensate having an average degree of polycondensation of less than 10 is obtained, which upon further heating is converted into a powder. This powder is subjected to a solid phase polycondensation process in an inert gas atmosphere. The process is based on a two-step procedure, the first step comprising the degradation of the scrap polymer into a low-molecular product, and the second step comprising the polycondensation of said low-molecular product to give a high-molecular product.

Brief Summary Text (13):

b) melting the shredded mixture so as to form a molten material,

Brief Summary Text (14):

c) optionally filtering the molten material so as to remove impurities having a particle size of equal to/greater than about 5 to 10 microns, so as to produce a filtered melt,

Brief Summary Text (15):

d) increasing by about 12 to about 150% as compared to the initial molecular weight of the filtered $\underline{\text{melt}}$, the average molecular weight of the filtered $\underline{\text{melt}}$, by post-condensation so as to produce a condensed mixture,

Brief Summary Text (17):

f) optionally feeding the granulated mixture to an <u>extruder and extruding</u> it together with virgin polyester so as to produce an <u>extruded</u> article.

Brief Summary Text (20):

b) melting the shredded mixture so as to form a molten material,

Brief Summary Text (21):

c) optionally filtering the molten material so as to remove impurities having a particle size of equal to/greater than about 5 to 10 microns, so as to produce a filtered melt,

Brief Summary Text (22):

d) solidifying the filtered melt, so as to produce a solidified mixture,

Brief Summary Text (25):

g) optionally feeding the condensed granulated mixture to an <u>extruder and extruding</u> it together with virgin polyester so as to produce an extruded article.

Detailed Description Text (11):

The process of the invention will be described in more detail below, with reference to the FIGURE. The diagrammatic representation below illustrates the working-up and recovery of PET waste materials according to the process of the invention. The diagram includes the individual process steps of: production--distribution--collection--comminution--working-up--melting post-condensation--granulating--new raw material. These preferred process steps are hereinafter described.

Detailed Description Text (16):

Biaxially oriented polyester films can also be reclaimed. In general such films have a RSV value of between 0.65 dl/g (SV about 790) and 0.80 dl/g (SV about 1,000), whereby high RSV values are achieved if the polymer raw material is dried well in vacuo prior to being processed into a film. This reduces the chain degradation of the polyester by hydrolysis during the processing into film. Care should also be taken that no substantial polymer chain degradation takes place due to excessive shearing forces which may arise during extrusion.

Detailed Description Text (22):

4. Comminution

Detailed Description Text (23):

It is generally preferred to transport, clean, and sort the waste material in the <u>comminuted</u> state. Accordingly, the waste material is first shredded and then cleaned. In special cases, for example when labels, lids or peel-off laminates have first to be removed from plastic containers, it is expedient not to shred the material until after the cleaning step.

Detailed Description Text (26):

<u>Washing</u> with water to remove dirt, <u>washing</u> with soda lye to remove paper labels, washing with hydrocarbon to remove adhesive layers and other coatings;

Detailed Description Text 6. Melting

Detailed Description Text (31):

The worked-up, shredded PET scrap material is <u>melted</u>. Prior to the <u>melting</u> process, by which the worked-up PET material is subjected to considerable thermal strain, the material preferably is <u>dried</u>, for example, in vacuo, in order to prevent or at least to minimize a possible chain degradation of the polyester due to hydrolysis during the further processing. The shredded material is <u>melted</u> and freed by filtering from impurities down to a particle size of 5 to 10 .mu.m.

Detailed Description Text (33):

The filtered <u>melt</u> is either post-condensed in the molten state or is first solidified and then granulated for a subsequent solid phase post-condensation.

Detailed Description Text (34):

The <u>melt</u> post-condensation is preferably performed continuously in a devolatizer equipped with a cage agitator, at a temperature above the <u>melting</u> point of PET, in the range of 270.degree. to 310.degree. C., preferably of about 280.degree. to 295.degree. C. In the course of the <u>melt</u> postcondensation the molecular weight of the filtered <u>melt</u> is increased by 12 to 150% compared to the initial molecular weight of the filtered <u>melt</u>. Preferably a vacuum of 0.5 to 5 mbar is applied, and the dwell time of the <u>melt</u> in the devolatizer is 1 to 4 hours. The conditions are selected depending on the desired increase in the molecular weight, expressed in terms of the SV value. For example, a reduced pressure of 1.5 to 2 mbar and a treatment time of 0.5 to 1.5 hours at 280 to 295.degree. C have to be selected for increasing the SV value from about 800 to 1050, and a reduced pressure of 3 to 5 mbar and a dwell time of 1 to 2 hours have to be selected for increasing the SV value from about 800 to 950. The granulated <u>melt</u> post-condensate is preferably fed into an <u>extruder and extruded</u> together with the virgin raw material to give cast films or other cast or molded articles.

Detailed Description Text (35):

If the filtered <u>melt</u> is first granulated and then subjected to a solid phase post-condensation, granules of relatively uniform size and shape are required, since the post-condensation is achieved via a diffusion process and is appropriately controlled. If the shape and size of the granules differ strongly, the molecular weight of the product obtained as a result of the solid phase postcondensation varies within a wide range.

Detailed Description Text (36):

The solid phase post-condensation is, for example, carried out continuously in a fluidized bed apparatus, employing an inert gas, such as nitrogen, as flushing gas and heat transfer agent. For increasing the SV value from about 800 to 1050, the temperature is adjusted to 210.degree. to 230.degree. C., and the dwell time is about 8 hours.

Detailed Description Text (37):

It is also possible to run the solid phase postcondensation discontinuously in a tumbler drier. A reduced pressure of about 0.1 mbar is then applied, and the reaction time is about 12 hours. The temperature is in the range of 225.degree. to 235.degree. C., and the SV value is increased from about 800 to 1050. A discontinuous solid phase condensation is particularly recommended in cases where the plastics material to be reclaimed has strongly differing initial property values and is present in varying amounts. Due to the high variability of the discontinuous solid phase condensation, this process is particularly advantageous if waste materials having varying molecular weights, pigment and other additives are to be treated. Generally, solid phase condensation is preferred if a reduced content of oligomers, acetaldehyde, terminal carboxyl groups as well as a high

clearness of the reclaimed material are desired.

CLAIMS:

- 1. A process for reclaiming thermally strained polyester waste materials comprising the steps of:
- a) shredding a mixture of polyester waste materials, said mixture comprising polyesters of different molecular weights and different degrees of conversion, so as to form a shredded polyester mixture,
- b) melting said shredded mixture so as to form a molten material,
- c) filtering said molten material down to a particle size of about 5 to 10 microns, by removing those impurities having a particle size of equal to about 5 to 10 microns and removing those impurities having a size of greater than 5 to 10 microns, so as to produce a filtered melt,
- d) increasing by about 12 to about 150% as compared to the initial molecular weight of said filtered <u>melt</u>, the average molecular weight of said polyesters, by post-condensation, so as to produce a condensed mixture, and
- e) recovering said condensed mixture.
- 3. A process of claim 2, additionally comprising the step of:
- f) feeding said recovered mixture to an extreuder and <u>extruding</u> it together with virgin polyester so as to produce an extruded article.
- 8. A process of claim 1, wherein said step d) is performed continuously with agitation and degassing at a reduced pressure and a temperature above the <u>melting</u> point of said polyesters.
- 12. A process of claim 1 additionally comprising between steps c) and d) the steps of:
- i) solidifying said filtered melt, so as to produce a solidified mixture,
- ii) granulating said solidified mixture so as to produce a granulated mixture, and
- iii) increasing by about 30 to 150%, as compared to the initial molecular weight of said granulated mixture, the average molecular weight of said granulated mixture.
- 13. A process of claim 12, additionally comprising the step of:
- f) feeding said granulated mixture to an <u>extruder and extruding</u> it together with virgin polyester so as to produce an <u>extruded</u> article.
- 19. A process of claim 12, wherein said step d) is performed continuously in a fluidized bed apparatus, using inert gases as $\underline{\text{heat}}$ transfer agents and flushing gases.
- 23. A process of claim 22, wherein step d) is performed in a tumbler <u>drier</u> at a temperature of about 200 to about 250.degree. C.